

## SPECIFICATION

### TITLE

“MARKER FOR USE IN A MAGNETIC ANTI-THEFT SECURITY SYSTEM”

### CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of U.S. Serial No. 10/371,894, filed February 21, 2003, which was a continuation of U.S. Serial No. 09/269,490, filed June 8, 1999, which was a National Stage Application under 37 CFR 371 of PCT/DE98/01984, filed July 15, 1998, which claimed priority from German 197 32 872.5, filed July 30, 1997.

### BACKGROUND OF THE INVENTION

The present invention is directed to a marker for use in a magnetic anti-theft security system. The marker is of a type composed of an oblong alarm strip composed of an amorphous ferromagnetic alloy, and at least one activation strip composed of a semi-hard magnetic alloy.

Magnetic anti-theft security systems and markers for security systems of the above type are well known and are described in detail in, for example, EP 0 121 649 B1 and WO 90/03652. First, there are magneto-elastic systems wherein the activation strip serves for activation of the alarm strip by magnetizing it; second, there are harmonic systems wherein the activation strip, after being magnetized, serves for the deactivation of the alarm strip.

The alloys with semi-hard magnetic properties that are employed for the pre-magnetization strip include Co-Fe-V alloys, which are known as VICALLOY, Co-Fe-Ni alloys, which are known as VACUZET, as well as Fe-Co-Cr alloys. These known semi-hard magnetic alloys contain high cobalt parts, some at least 45 weight %, and are correspondingly expensive.

In addition, while in their magnetically finally annealed condition, these alloys are brittle, so that they do not exhibit adequate ductility in order to adequately meet the demands given markers or display elements for anti-theft security systems. One important

demand, namely, is that these activation strips should be insensitive to bending or deformation.

In the meantime, a switch has been made to introduce the markers of the anti-theft security systems directly into the product to be secured (source tagging). Such source tagging imposes the additional demand that the semi-hard magnetic alloys should be able to be magnetized from a greater distance or with smaller fields. To satisfy this additional demand, it has been shown that the coercive force H must be limited to values of, at most, 24 A/cm.

On the other hand, however, an adequate opposing field stability is also required, which determines the lower limit value of the coercive force. Only coercive forces of at least 10 A/cm are thereby suited.

Further, the remanence should be optimally slight under bending or tensile strength. A change of less than 20% is prescribed as a guideline.

#### SUMMARY OF THE INVENTION

It is an object of the present invention to provide a marker of the above-described type for a magnetic anti-theft system, having an activation strip which satisfies the above demands for source tagging.

This object is inventively achieved in a marker having an activation strip composed of a semi-hard magnetic alloy comprising 8 to 25 weight % nickel, 1.0 to 4.5 weight % aluminum, 0.5 to 3 weight % titanium and the balance iron.

In a preferred embodiment of the invention, the content of aluminum is between 1.2 and 2.8 weight %. Optimum results are achieved with a content of aluminum between 1.5 and 2.8 weight %.

For best results, the content in weight % of nickel, aluminum and titanium should satisfy the following formula:

$$35 \leq \text{Ni}(1,75\text{Al} + \text{Ti}) \leq 110, \text{ preferably } 40 \leq \text{Ni}(1,75\text{Al} + \text{Ti}) \leq 90.$$

The alloy can further contain 0 to 5 weight % cobalt and/or 0 to 3 weight % molybdenum or chromium and/or at least one of the elements Zr, Hf, V, Nb, Ta, W, Mn, Si in individual parts of less than 0.5 weight % of the alloy and in an overall part of less than 1 weight % of the alloy and/or at least one of the elements C, N, S, P, B, H, O in individual parts of less than 0.2 weight % of the alloy and in an overall part of less than 1 weight % of the alloy.

The alloy is characterized by a coercive strength  $H_c$  of 10 to 24 A/cm and a remanence  $B_r$  of at least 1.3 T (13,000 Gauss).

The inventive alloys are highly ductile and can be excellently cold-worked before the annealing, so that cross-sectional reductions of more than 90% are also possible. An activation strip having a thickness of less than 0.05mm can be manufactured from such alloys, particularly by cold rolling. In addition, the inventive alloys are characterized by excellent magnetic properties and resistance to corrosion.

A preferred alloy is a semi-hard magnetic iron alloy according to the present invention that contains 13.0 to 17.0 weight % nickel, 1.8 to 2.8 weight % aluminum as well as 0.5 to 1.5 weight % titanium. By reducing the aluminum content, the magnetostriction can, in particular, be especially favorably set.

Typically, the activation strips are manufactured by melting the alloy under a vacuum and then casting to form an ingot. Subsequently, the ingot is hot-rolled into a tape or ribbon at temperatures above 800°C, then intermediately annealed at a temperature above 800°C and then rapidly cooled. A cold working, expediently cold rolling to provide a cross-sectional reduction of approximately 90% is followed by an intermediate annealing at approximately 700°C. A cold working, expediently cold rolling to provide a cross-sectional reduction of at least 60% and preferably 75% or more subsequently occurs. As a last step, the cold-rolled tape or ribbon is annealed at temperatures from approximately 400°C to 600°C. The activation strips are then cut to length.

Other advantages and features of the invention will be readily apparent from the following description, the claims and drawings.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates the demagnetization behavior of the inventive Fe-Ni-Al-Ti alloys after an alternating field magnetization at 4 A/cm, dependent on the coercive force  $H_c$ ;

Fig. 2 illustrates the demagnetization behavior of the inventive Fe-Ni-Al-Ti alloys after an alternating field magnetization at 20 A/cm, dependent on the coercive force  $H_c$ ;

Fig. 3 illustrates the change of the remanence  $B_r$  under tensile stress of two embodiments of the inventive alloy, compared to a prior art alloy; and

Fig. 4 illustrates the relative change of the magnetic flux, in percent, at various coercive field strengths after mechanical deformation for an embodiment of an inventive alloy compared to a prior art alloy.

#### DESCRIPTION OF THE PREFERRED EMBODIMENTS

The following demands derive for the suitability of an alloy for an activation strip in an anti-theft security system, particularly for a system employing source tagging:

The change of the remanence under bending or tensile stress should be optimally slight. A change of 20% is prescribed as a guideline. As can be seen from Fig. 3, values  $\leq 10\%$  are achieved with the alloys of the present invention.

It can be seen from Fig. 4 that, in addition to being determined by the alloy, the coercive field strength and the bending radius also determine the change of the flux. Given corresponding coercive field strengths, the alloys according to the present invention achieve values  $< 5\%$  given bending radii  $\geq 12\text{mm}$  or, respectively, values  $< 10\%$  given bending radii  $\geq 4\text{mm}$  and thicknesses of approximately  $50\mu\text{m}$ .

The relationship of the saturation at a given, slight magnetizing field strength of, for example, 40 A/cm to the saturation  $B_f$  given a magnetic field in the kOe range should be nearly 1, which can be seen from Fig. 3.

The opposing field stability should be of such a nature that the remanence  $B_s$  still retains at least 80% of its original value after an opposing field magnetization of a few A/cm.

Finally, the remanence should retain only 20% of the original value after a demagnetization cycle with a predetermined magnetic field.

In detail, this means that a magnetization of the activation strip, i.e., an activation/deactivation of the marker or display element, can also occur on site. However, only very small fields are generally available there. The saturation that is achieved should differ only slightly from the value given high magnetizing fields in order to guarantee identical behavior of the marker or display elements.

The display elements or markers must be of such a nature that their remanence  $B_r$  changes only slightly in the proximity of the coils in the detection locks as a consequence of a field that is elevated thereat and is potentially oriented in the opposite direction. As can be seen from Fig. 1, the inventive alloys exhibit an opposing field stability as demanded.

Finally, the markers or display elements must be capable of being demagnetized with relatively small fields, i.e., deactivated given magneto-elastic markers or, respectively, activated given harmonic display elements or markers. Figure 2 illustrates these relationships given the inventive alloys.

Simultaneously, meeting these last three demands yields extremely great limitations for the accessible ranges of the coercive forces  $H_c$ , since the three demands are contradictory.

The alloys of the present invention are typically manufactured by casting a melt of the alloy constituents in a crucible or furnace under a vacuum or a protective gas atmosphere. The temperatures thereby lie at approximately 1600°C.

The casting typically utilizes a round ingot mold. The cast ingots of the present alloys are then typically processed by hot working, intermediate annealing, cold working and a further intermediate annealing. The intermediate annealing is performed for the purpose of homogenization, grain sophistication, shaping or the creation of desirable mechanical properties, particularly a high ductility.

An excellent structure is achieved, for example, by the following process:

Thermal treatment at, preferably, temperatures above 800°C, rapid cooling and annealing. Preferred annealing temperatures lie at 400°C through 600°C, and the annealing times typically lie advantageously between one minute through 24 hours. A cold working corresponding to a cross-sectional reduction of at least 60% before the annealing is, in particular, possible with the inventive alloys.

The coercive force and the rectangularity of the magnetic B-H loop are enhanced by the step of annealing, and this is implemented for the demands made of the activation strips.

The manufacturing method for especially good activation strips comprises the following steps:

- 1) Casting at 1600°C.
- 2) Hot rolling of the ingot at a temperature above 800°C.
- 3) Multi-hour intermediate annealing at about 800°C with quenching in water.
- 4) Cold rolling corresponding to a cross-sectional reduction of approximately 90%.
- 5) Intermediate annealing at approximately 700°C.
- 6) Cold working corresponding to a cross-sectional reduction of approximately 90%.
- 7) Multi-hour intermediate annealing at approximately 700°C.
- 8) Cold working to produce a cross-sectional rejection of approximately 70%.

- 9) Multi-hour annealing at approximately 480°C.
- 10) Cutting and trimming the activation strips.

Activation strips that exhibited an excellent coercive force  $H_c$  and a very good remanence  $B_r$  were manufactured with this method. The magnetization properties and the opposing field stability were excellent.

The manufacture of several embodiments of Fe-Ni-Al-Ti activation strips in accordance with the invention is described in detail on the basis of the following examples:

Example 1:

An alloy with 18.0 weight % nickel, 3.8 weight % aluminum, 1.0 weight % titanium and the balance iron was melted under a vacuum. The resulting ingot was hot-rolled at approximately 1000°C, intermediately annealed for one hour at 1100°C and rapidly cooled in water. After a subsequent cold-rolling with a cross-sectional reduction of 80%, the resulting ribbon was again intermediately annealed for one hour at 1100°C and rapidly cooled in water. After a further cold working with a cross-sectional reduction of 50%, the ribbon was intermediately annealed for four hours at 650°C. To provide a cross-sectional reduction of 90%, the ribbon was subsequently cold-rolled and annealed at 520°C for three hours and then cooled in air. A coercive force  $H_c$  equal to 23 A/cm as well as a remanence  $B_r$  equal to 1.48T were measured.

Example 2:

An alloy with 15.0 weight % nickel, 3.0 weight % aluminum, 1.2 weight % titanium and balance iron was processed as in Example 1 but with the last intermediate annealing at 700°C, the last cold working provided a cross-sectional reduction of 70% as well as a final annealing was at 500°C. A coercive force  $H_c$  equal to 21 A/cm and a remanence  $B_r$  equal to 1.45T were measured.

Example 3:

An alloy with 15.0 weight % nickel, 3.0 weight % aluminum, 1.2 weight % titanium and balance iron was manufactured as in Example 2. Deviating therefrom, the last

intermediate annealing occurred at 650°C, the last cold working to provide a cross-sectional reduction of 85% and the annealing treatment was at 480°C. A coercive force  $H_c$  equal to 20 A/cm and a remanence  $B_r$  equal to 1.53T were measured.

Example 4:

An alloy with 15.0 weight % nickel, 3.0 weight % aluminum, 1.2 weight % titanium, 2.0 weight % molybdenum and balance iron was manufactured as in Example 2. After an annealing treatment at 480°C, a coercive force  $H_c$  equal to 20 A/cm and a remanence  $B_r$  equal to 1.56T were measured.

Example 5:

An alloy with 15.0 weight % nickel, 3.0 weight % aluminum, 0.8 weight % titanium and balance iron was melted under a vacuum. The resulting ingot was hot-rolled at approximately 1000°C, intermediately annealed at 900°C for one hour and rapidly cooled in water. After a following cold-rolling with a cross-sectional reduction of 90%, the resulting ribbon was intermediately annealed for four hours at 650°C. To produce a cross-sectional reduction of 95%, the tape was subsequently cold-rolled and annealed for three hours at 460°C and then air-cooled. A coercive force  $H_c$  equal to 14 A/cm and a remanence  $B_r$  equal to 1.46T were measured.

Example 6:

An alloy with 15.0 weight % nickel, 2.5 weight % aluminum, 1.2 weight % titanium and balance iron was manufactured as in Example 5, but with a cross-sectional reduction of 83% and an annealing treatment at 420°C. A coercive force  $H_c$  equal to 17 A/cm and a remanence  $B_r$  equal to 1.44T were measured.

Example 7:

An alloy with 20.0 weight % nickel, 1.0 weight % aluminum, 1.2 weight % titanium and the balance iron was melted under a vacuum. The resulting ingot was hot-rolled at approximately 1000°C, intermediately annealed for one hour at 1100°C and rapidly cooled in water. After a subsequent cold-rolling with a cross-sectional reduction of 80%, the resulting ribbon was again intermediately annealed for one hour at 1100°C and rapidly cooled



in water. After a further cold working with a cross-sectional reduction of 50%, the ribbon was intermediately annealed for four hours at 650°C. To provide a cross-sectional reduction of 75%, the ribbon was subsequently cold-rolled and annealed at 450°C for three hours and cooled in air. A coercive force  $H_c$  equal to 13.4 A/cm as well as a remanence  $B_r$  equal to 1.35T were measured.

Example 8:

An alloy with 15.0 weight % nickel, 1.3 weight % aluminum, 0.6 weight % titanium and the balance iron was melted under a vacuum. The resulting ingot was hot-rolled at approximately 1000°C, intermediately annealed for one hour at 1100°C and rapidly cooled in water. After a subsequent cold-rolling with a cross-sectional reduction of 80%, the resulting ribbon was again intermediately annealed for one hour at 1100°C and rapidly cooled in water. After a further cold working with a cross-sectional reduction of 50%, the ribbon was intermediately annealed for four hours at 660°C. To provide a cross-sectional reduction of 85%, the ribbon was subsequently cold-rolled and annealed at 550°C for three hours and cooled in air. A coercive force  $H_c$  equal to 17.3 A/cm as well as a remanence  $B_r$  equal to 1.31T were measured.

A satisfactory magnetization behavior and a usable opposing field stability are derived in all exemplary embodiments.

Although various minor modifications may be suggested by those versed in the art, it should be understood that we wish to embody within the scope of the patent granted hereon all such modifications as reasonably and properly come within the scope of our contribution to the art.